

Development of SiC-based Gas Sensors for Aerospace Applications

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ABSTRACT

Silicon carbide (SiC) based gas sensors have the ability to meet the needs of a range of aerospace applications including leak detection, environmental control, emission monitoring, and fire detection. While each of these applications require that the sensor and associated packaging be tailored for that individual application, they all require sensitive detection. The sensing approach taken to meet these needs is the use of SiC as a semiconductor in a Schottky diode configuration due to the demonstrated high sensitivity of Schottky diode-based sensors. However, Schottky diode structures require good control of the interface between the gas sensitive metal and SiC in order to meet required levels of sensitivity and stability. Two examples of effort to better control the SiC gas sensitive Schottky diode interface will be discussed. First, the use of chrome carbide as a barrier layer between the metal and SiC is discussed. Second, we report the first use of atomically flat SiC to provide an improved SiC semiconductor surface for gas sensor deposition. An example of the demonstration of a SiC gas sensor in an aerospace applications is given. It is concluded that, while significant progress has been made, the development of SiC gas sensor systems is still at a relatively early level of maturity for a number of applications.

INTRODUCTION

Silicon carbide (SiC) has high potential as the electronic semiconductor material for a new family of high temperature sensors and electronics. Silicon carbide can operate as a semiconductor in conditions under which silicon cannot adequately perform, such as at temperatures above 400°C or in corrosive environments. [1]. One area where SiC semiconductor technology can be applied is in chemical sensing. Silicon carbide gas sensors have been in development for a number of years using a range of designs including capacitors [2], transistors [3], and Schottky diodes [4-7]. These sensors have been shown to be highly sensitive to several gases, including hydrogen and hydrocarbons, making them useful for a range of applications.

A range of aerospace applications require chemical sensing technology [8]. One application area is the monitoring of fuel leaks in launch vehicles. Detection of low concentrations of hydrogen and hydrocarbon fuels is critical in avoiding explosive conditions that could harm personnel and damage the vehicle. Reliable vehicle operation also depends on the timely and

accurate measurement of these leaks. Detection of low concentrations of fuel, such as RP-1 jet fuel, is generally necessary from room temperature to cryogenic ambient and in air or inert gas atmospheres. Further, measurement of highly toxic propellants such as hydrazine in concentrations as low as the parts per billion (ppb) level is desired, especially for manned systems such as the International Space Station.

A second application area is the monitoring of emissions from high temperature environments such as combustion systems or chemical reactors. In both aeronautic and commercial combustion emissions monitoring applications, sensitive detection of hydrocarbons can be used to reduce emissions and potentially monitor the efficiency and health of the engine. For in-situ engine monitoring, high temperature operation is necessary. A significant challenge in this application is determining the relative hydrocarbon concentration in a mixed and varying chemical environment.

A third application area is fire detection on commercial aircraft and space vehicles. Rapid detection and location determination of a fire is extremely important to avoid catastrophic situations. While existing smoke detectors are able to detect fires, they have a high incidence of false alarms and thus a second, independent method of fire detection to complement conventional smoke detection techniques is required. The measurement of chemical species indicative of a fire is envisioned to help reduce false alarms and improve system safety. While carbon monoxide (CO) and carbon dioxide (CO₂) are important, concentrations of other gases such as hydrocarbons are also of interest.

These applications require operation in a variety of challenging conditions: from cryogenic temperature to above 600°C, from chemically inert environments to highly corrosive engine conditions, and from the detection of one gas over a wide concentration range in inert environments to the detection of several gases over more narrow concentration ranges in the presence of interfering gases. These applications commonly require high sensitivity, long-term stability, good repeatability, and usually sensor operation at elevated temperatures to detect the gases of interest. The combination of these sensor requirements has led NASA Glenn Research Center (NASA GRC) SiC gas sensor development to concentrate on sensors based on the Schottky diode structure.

A Schottky diode is composed of a metal in direct contact with a semiconductor (MS) or a metal in contact with a very thin insulator or oxide on a semiconductor (MIS or MOS). For gas sensing applications, the metal is often catalytic. The advantage of Schottky diode gas sensors are their high sensitivity. The detection mechanism for hydrogen (H₂) involves the dissociation of H₂ on the surface of a catalytic metal leading to the formation of a dipole layer at the interface of the metal and the insulator (or metal-semiconductor interface depending on the structure). This dipole layer affects the effective Schottky barrier height of the diode resulting in an exponential change in the forward current and a quadratic change in the capacitance [9-10] while the diode is under fixed bias. The detection of hydrocarbons is possible if the sensor is operated at a high enough temperature to dissociate the hydrocarbon and produce atomic hydrogen. The resulting atomic hydrogen affects the sensor output in the same way as molecular hydrogen [4, 11-12]. Predominately, the temperature for sensitive hydrocarbon detection is beyond the upper limit for silicon-based Schottky diode functionality and thus SiC enables high temperature detection of hydrocarbons with exponential sensitivity.

The successful use of the Schottky diode structure depends on strict control of the metal-semiconductor interface. One complicating factor in control of this interface is the operation of gas sensors at high temperatures. Higher temperature operation implies possible reactions,

especially metal silicide formation, between the catalytic sensing metal and the SiC. While one can decrease the metal/SiC reactivity, if this occurs in such a manner so as to “pin” the interface potential barrier, then sensor sensitivity can be significantly decreased [7]; effectively defeating the purpose of using a Schottky diode structure. Thus, the choice of surface treatment or barrier layer(s) between the catalytic metal and the SiC substrate is complicated by simultaneous requirements of high sensor stability during high temperature operation while maintaining high sensitivity.

A second major complicating factor in the control of the SiC interface is the nature of the SiC itself. Compared to silicon wafer standards, present-day SiC wafers are small, expensive, and of inferior crystalline quality. In addition to high densities of extended crystalline defects such as micropipes and closed-core screw dislocations, commercial SiC wafers also exhibit significantly rougher surfaces, and larger warpage than is typical for silicon wafers [13]. The highly variable SiC surface itself significantly complicates efforts to control the catalytic metal/SiC interface. Despite these obstacles, significant progress has been made in the development and use of SiC Schottky diode gas sensors.

The purpose of this paper is to discuss the development and application of SiC Schottky diode gas sensors. Two examples will be given related to NASA GRC efforts to control the interface: 1) The use of chrome carbide as a barrier layer between the catalytic metal and the SiC semiconductor; and 2) The first use to our knowledge of on-axis and atomically flat SiC as the semiconductor surface for gas sensor fabrication. A demonstration of the use of the chrome carbide barrier approach in an aerospace application will also be presented. It is concluded that SiC Schottky diode gas sensors have use in a range of applications presently and continue to show promise because of their potential high temperature operation, high sensitivity, and the ability to integrate the sensors with on-chip high temperature electronics. However, their full potential is just beginning to be explored.

EXPERIMENTAL

The characterization of two types of sensors will be discussed in this paper. The first type is a Pt/Cr₃C₂/SiC Schottky diode. The second is a sensor containing Pt/SiC on both atomically flat and non-atomically flat mesas side-by-side on the same chip. Backside contacts for both samples were achieved by E-beam evaporation of nickel (Ni) at a thickness of ~5000 Å followed by an anneal at 1000°C for 5 min in N₂. The Pt/Cr₃C₂/SiC diodes were formed on a commercially available 3-5° off-axis 6H-SiC substrate with 0.3 μm thick epilayer of doping of 2.7 E+16. Sputtering was used to deposit 600 angstroms (Å) of Cr₃C₂ followed by 300 Å of Pt. A lift-off technique was then used to form circular Pt/Cr₃C₂/SiC Schottky patterns of diameter 830 μm.

Atomically flat or step-free surfaces were produced on commercially purchased on-axis 6H-SiC wafers by first dry reactive ion etching (RIE) trenches into the wafer surface to form an array of isolated growth mesas. Following an in-situ pre-growth etch at 1530°C for 15 minutes, pure stepflow epitaxial growth is then used to grow all initial surface steps out to the edge of the mesa leaving behind a mesa with a topmost surface that is step-free [14]. Only mesas that do not contain screw dislocations can be rendered step-free (i.e. atomically flat) since steps cannot be removed from a mesa threaded by screw dislocations because the screw dislocations continuously provide kinetic growth steps to the surfaces of such mesas. Step-free mesas have

characteristic cantilever development that can be observed with optical microscopy which was used for identifying device mesas that were step-free [15]. The growth was carried out in a modified, commercial, horizontal flow, chemical vapor deposition (CVD) system with an uncoated susceptor. The growth was performed at a pressure of 200 mb, and a temperature of 1530°C (+/- 20°C). The precursors used were C₃H₈ as the carbon source, and SiH₄ as the silicon source, in a hydrogen carrier gas with a Si/C ratio of 1.8. The growth time was 30 minutes. Standard patterning and lift off techniques were combined with the sputtering of 300 Å of Pt to form Pt/SiC diodes both on atomically flat and non-flat mesas.

Figure 1 shows the two side-by-side mesas used for testing in this experiment where one was step-free or atomically flat while the second was not due to screw dislocations. The two sensors were tested with a contact on each sensor and the data was taken for both sensors simultaneously. It should be noted that both of these samples are on-axis in contrast to standard SiC surfaces (typically 3-8 degrees off of the (0001) crystallographic basal plane) on which all SiC gas sensors have previously been fabricated.

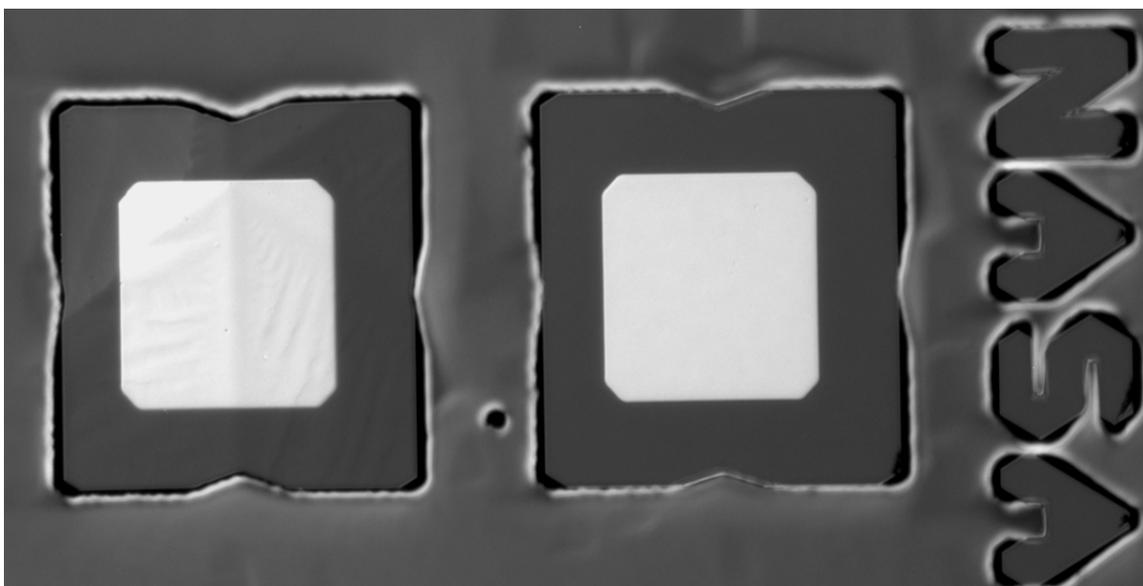


Figure 1. The two samples tested in this paper: The left hand side is the non-atomically flat sensor and the right hand side is the atomically flat sample. Surface differences between the two sensors are noticeable, such as hillock morphology from a screw dislocation on the left mesa.

The gas sensor testing facility and sample connections have been described elsewhere [4]. The sample rested on a hot stage whose temperature was controlled from room temperature to near 600°C. Current-time (I-t) measurements were taken to characterize diode responses as a function of time during exposure to a variety of gases, and current-voltage (I-V) measurements were taken to characterize the diode's electronic properties in given gas environment. The gain of the sensor was calculated as the difference in currents between hydrogen bearing gas and air then divided by the air values, i.e., the change in signal divided by the baseline. Surface and interface of the samples were analyzed by Auger Electron Spectroscopy (AES) using a PHI model 590A. Data was collected using a 5 keV electron beam, 1.3 A beam current, and depth profiled at a sputter rate of 30Å/min using a 4 keV argon ion gun.

RESULTS AND DISCUSSION

Motivation for Specific Sensor Structures

Previous results suggested that PdCr can be a stable system (PdCr/SiC) that has high sensitivity to hydrogen [5]. The results of surface and interface analyses indicated that after testing (or heating), a part of the PdCr thin film was still free of silicide and the sensor surface was also relatively clean (lower silicon oxides content) compared to the Pd/SiC sensors examined [16]. It was suggested that a carbide component at the interface region may be critical in maintaining sensor performance stability and sensitivity. This carbide was thought to form through an interfacial reaction in the early stages of heating of the PdCr/SiC interface. However, despite these promising results, the ability to systematically produce films with combined stability and sensitivity has proven problematic [7]. One potential reason for this is the lack of consistent control of the interface between the catalytic metal and the SiC. This paper will present two examples of efforts to better control the catalytic metal/SiC interface: 1) Directly forming a chrome carbide barrier layer by depositing a layer of Cr_3C_2 between the metal and SiC. The metal used in this example is Pt for an application related to hydrocarbon detection; and 2) Controlling the surface of the SiC by use of on-axis and atomically flat SiC. While further detailed discussion of these systems is planned in future publications, these examples give an indication of approaches to overcome interface issues associated with SiC Schottky diode gas sensors.

Barrier Layer: Pt/Cr₃C₂/SiC Structure

A Pt/Cr₃C₂/SiC Schottky diode structure using commercial off-axis SiC epilayer was tested at 450°C for 70 hours then 580°C for 600 hours for a total of 670 hours. The sensor was tested by first being exposed to air for 5 minutes, N₂ for 5 minutes, 0.5% propylene in N₂ for 10 minutes, pure N₂ for 5 minutes, and then air. At lower temperatures and voltages, the sensor had limited response but showed good response at 580°C with forward bias measurement voltages of 1.0V or 3.2 V. The test was stopped at 670 hours for sample analysis with the sensor viewed as stabilized and operational.

The propylene/air gain of the sensor over time at 580°C measured at these voltages is shown in the inset of Figure 2. The response varied with voltage exhibiting somewhat better stability at 3.2 V than 1 V (Figure 2 inset). While the sensor loses some sensitivity over the first 200 hours at 580°C, the sensor seems to stabilize after this break-in period. The sensor shows a consistently strong response to propylene with an average gain of over near two thousand for at least 270 hours. Figure 2 shows representative data comparing the sensor response at 3.2 V at 500 hours and 670 hours. The gas exposure period in Figure 2 is somewhat extended for the 670 hours data to allow better comparison between the two sets of data. The data shows good repeatability of signal in form and magnitude. The recovery in inert environments is slow but previously this has been found to be chamber dependent. The data suggests a sensor with very good sensitivity to propylene and reasonable stability at 580°C, even after a total of 600 hours at 580°C. While this sensor was sensitive to propylene (hydrocarbons), it was much less sensitive to hydrogen.

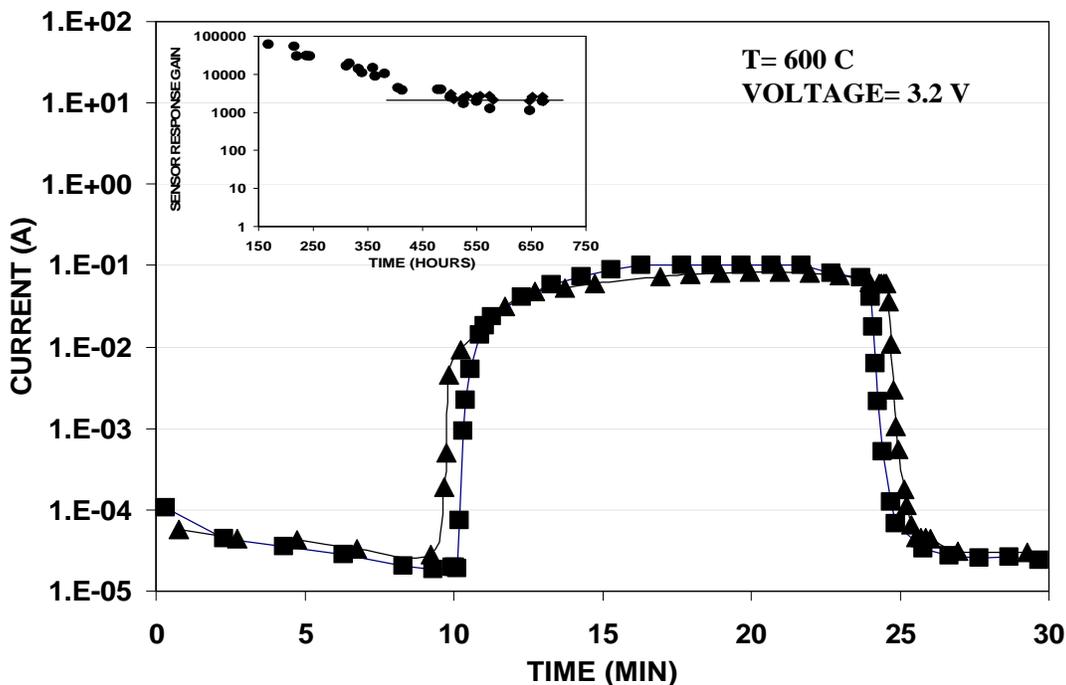


Figure 2. The Pt/Cr₃C₂/SiC sensor tested at 580°C at 500 hours (■) and 670 hours (▲) in 0.5% propylene. Inset: The sensor gain over time at 580°C measured at 1.0 V (●) and 3.2 V (◆).

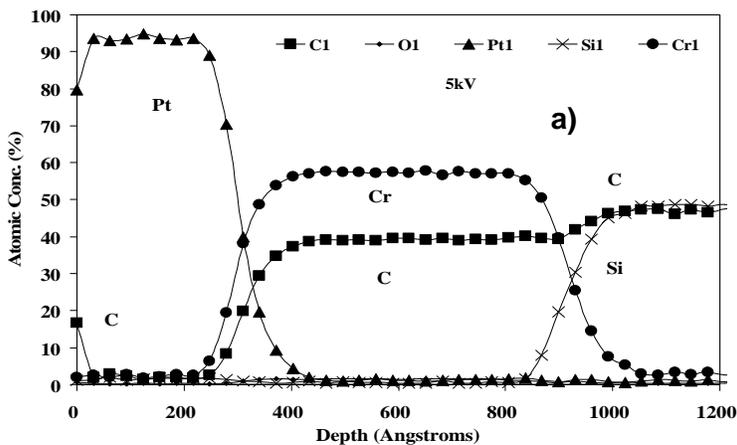
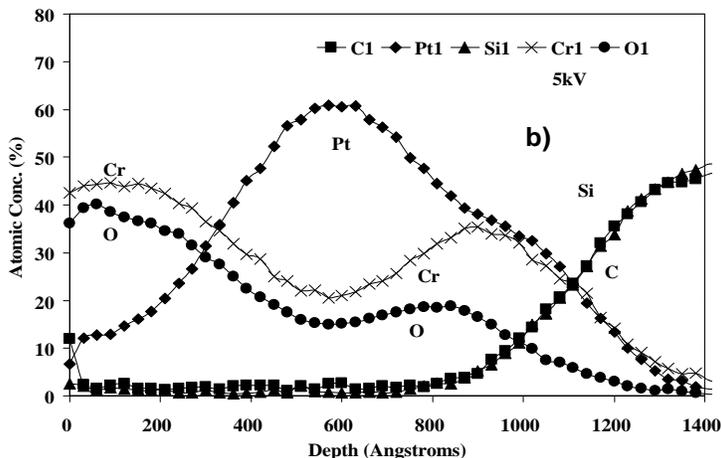


Figure 3. AES depth profile of the Pt/Cr₃C₂/SiC sensor:

a) as-deposited.

b) after annealing at 450°C for 70 hrs and then 580°C for 600 hours and periodic exposure to hydrogen bearing gases.



However, the sensor did change response over time from the onset of sensor testing. AES analysis was performed on Pt/Cr₃C₂/SiC samples both before and after testing in order to gain insight into the reason for the change. The Auger results from the as-deposited sample are shown in Figure 3a. The results show well-defined interfaces with no indication of Pt silicide (PtSi_x) formation throughout the sample. Figure 3b shows the AES analysis of the sensor after 670 hours of testing. The data did not show any evidence for carbide or carbon remaining from the original Cr₃C₂ layer, or even any sign of migration of carbide or carbon into the Pt layer. Oxygen had diffused into the layers, replacing the carbon to form an oxygen-deficient chromium oxide, which migrated toward the surface. This migration likely allowed the Pt to diffuse toward the SiC interface. It should be noted that massive formation of metal silicides [5,7,16], the likely cause of sensor failure in other sensor structures, was not observed in this sample.

Although the reaction mechanisms for the Pt layer on Cr₃C₂/SiC are still not completely understood, several observations can be made. The Cr₃C₂ layer appears to be effective in preventing immediate reaction of the Pt layers with silicon from the crystal to form metal silicides. The presence of carbide in the chromium layer slows down migration of the chromium to the surface, and allows formation of an oxygen-poor chromium oxide, which gradually diffuses throughout the metal layer. This chrome sub-oxide likely prevents metal silicide formation at the SiC interface.

Overall, the sensor showed stable operation with good sensitivity at 580°C. Improvements are necessary to decrease the break-in period and the long-term operation limit of this sensor design still remains to be determined. Nonetheless, a sensor with this high of a sensitivity combined with limited drift after a break-in period can be used for a number of hydrocarbon detection applications.

SiC Surface Treatment: Atomically Flat SiC

Two samples were tested side by side on the same chip: Pt/SiC Schottky diodes on atomically flat SiC (AF) and non-atomically flat SiC (NAF) as shown in Figure 1. This is the first reported gas sensor made on atomically flat SiC. A second major point to note is that both of these samples are also fabricated with on-axis SiC. The sensor is tested by first being exposed to air for 5 minutes, N₂ for 5 minutes, 0.5% propylene in N₂ for 10 minutes, pure N₂ for 5 minutes, and then air. The sensors were tested at 100°C for 44 hours, 200°C for 145 hours, and then at 300°C for near 500 hours. The sensors had nearly the same response at 100°C and 200°C. The test was stopped at near 690 hours for sample analysis with the sensor viewed as stabilized and operational. Figure 4 shows a comparison of the sensor gain in hydrogen between the AF and NAF samples at various temperatures from room temperature to 300°C.

The difference in response between these two sensors takes effect when the sensor is heated to 300°C and is easily evident in Figure 4. Figure 4 shows the data starting at 200°C and finishing at 300°C data. The AF sensor gain increases dramatically compared to the NAF sensor and stabilizes near 325 hours into the testing or after nearly 200 hours at 300°C. The gain of the AF sensor is near 7250 while that of the NAF sensor is near 50. Thus the gain of the AF sensor response averages nearly 145 times greater than that of the NAF sensor. While some difference in sensor response was expected, the dramatic difference between two sensors side-by-side on the same sensor chip was surprising.

The two mesas which hold the AF and NAF sensors were examined by AES to determine if there were chemical differences between the two sensors which could explain the difference in

sensor behavior. An as-deposited sample was also examined by AES. The interface between the Pt and the SiC shows approximately 50 Å of silicide formation on the as-received sample (not shown but see Figure 5). This layer is most likely formed during metal sputtering. Other than some expected carbon and oxygen contaminants on the surface, the profile showed clean metallization layers with very little reaction taking place. AES analysis of the tested, NAF Pt/SiC mesa was found to be almost identical to the AF mesa as shown in Figure 5, and both are very similar to the as-deposited sample. Other than the thin layer of silicide formation found at the interface due to the sputtering process, Auger cannot reveal any evidence for chemical reactions taking place, even after over 600 hours of testing. Therefore, chemical differences detectable by Auger cannot explain the very different electrical behavior of the two tested AF and NAF mesas.

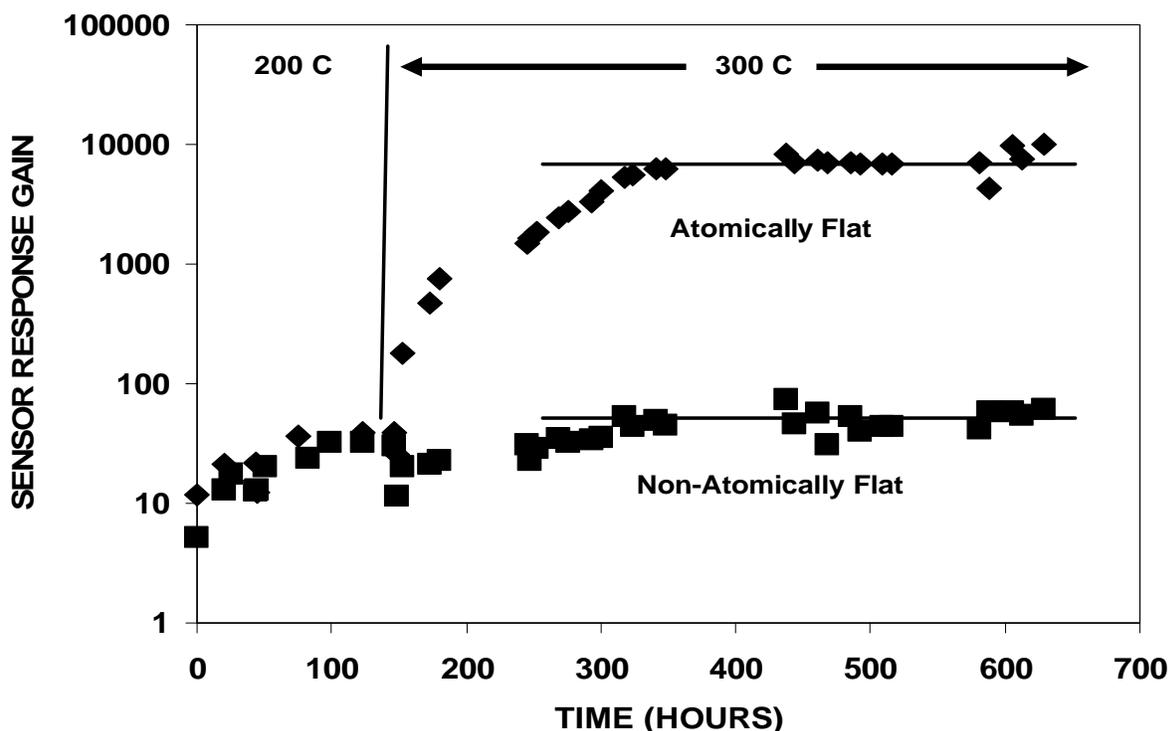


Figure 4. Comparison of sensor gain to 0.5% hydrogen between Pt/SiC sensors deposited on atomically flat SiC (◆) and non-atomically flat SiC (■). At near 150 hours of testing at 200°C, both sensors are heated to 300°C. The difference in response between the sensors is easily observed.

The Pt/SiC interface remains essentially as deposited on both sensors even after prolonged testing. From these results, it would appear that the differences in sensing properties that they each exhibit must be related to either electronic or structural interface differences between atomically flat and non-atomically flat SiC. These preliminary results suggest a strong advantage to the use of on-axis and atomically flat SiC over the standard materials on which SiC Schottky diode gas sensors are fabricated. A significant amount of work still needs to be done to better characterize the samples and determine the mechanisms and extent of advantages that the atomically flat and on-axis systems might provide.

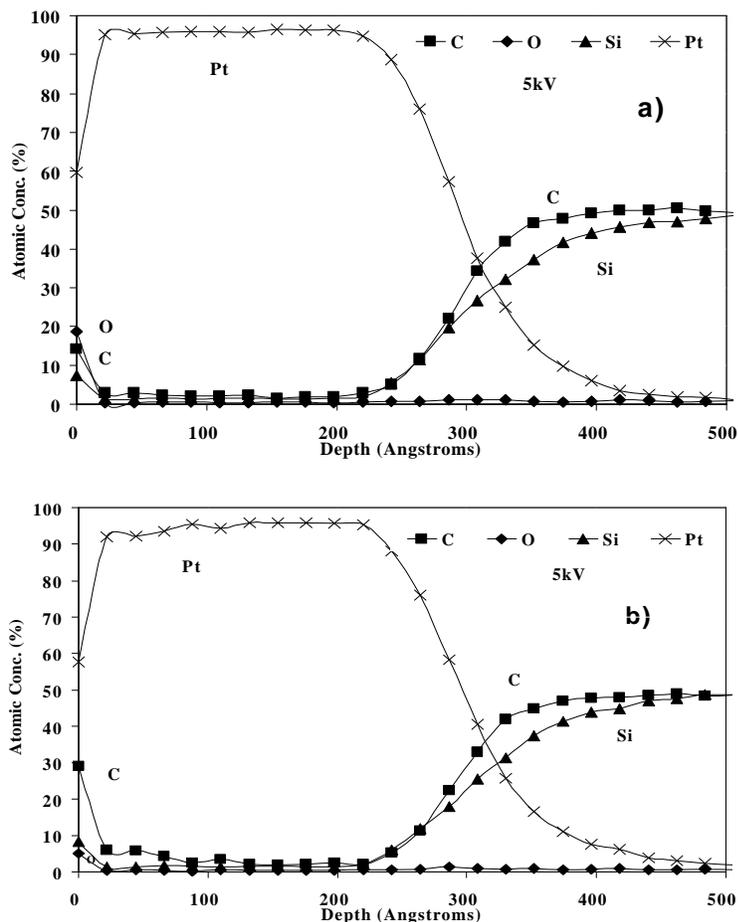


Figure 5. AES depth profile after testing, which included 500 hours at 300°C, of: a) atomically flat sensing film and b) non-atomically flat sensing film of the Pt/SiC Schottky diodes. The major feature of these figures is their similarity.

SENSOR APPLICATION

While work proceeds towards improving long-term sensitivity and high temperature durability of SiC Schottky diode gas sensors, these sensors are useful for a range of applications. One area of development is an integrated smart leak detection system for a range of propulsion systems. The objective is to produce a microsensor array, which includes hydrogen, oxygen, and hydrocarbon sensors by microfabrication (MEMS) based technology. Thus, a range of potential launch vehicle fuels (hydrogen or hydrocarbons) and oxygen can be measured simultaneously. The array is being incorporated with signal conditioning electronics, power, data storage, and telemetry. The final system will be self-contained with the surface area comparable to a postage stamp. Thus, this postage stamp sized “Lick and Stick” type gas sensor technology can enable a matrix of leak detection sensors placed throughout a region with minimal size and weight as well as with no power consumption from the vehicle. The sensors can detect a fuel leak from next

generation vehicles, and combine that measurement with a determination of the oxygen concentration to ascertain if an explosive condition exists. Sensor outputs are fed to a data processing station, enabling realtime visual images of leaks, and enhancing vehicle safety.

A prototype model of the “Lick and Stick” sensor system has been fabricated and is shown in Figure 6a. The complete system has signal conditioning electronics, power, data storage, and telemetry with hydrogen, hydrocarbon, and oxygen sensors. Figure 6b shows the operation of the electronics plus the three sensor system simultaneously. In particular, the data highlights the response of the SiC-based gas sensor at various hydrocarbon fuel (RP-1) concentrations. The hydrogen and oxygen sensors are not SiC-based and their operation is described elsewhere [8]. The oxygen concentration is held constant and the hydrogen sensor signal shows no response, suggesting a lack of cross-sensitivity between the hydrogen and hydrocarbon sensors to the detection of this hydrocarbon. Cross-sensitivity between the hydrocarbon and hydrogen sensor to other gases may be an issue depending on the application. The hydrocarbon sensor, a Pt/Cr₃C₂/SiC Schottky diode, is operated at 400°C, and is able to detect fuel concentrations from 300 ppm to 3000 ppm. The magnitude of the response to 300 ppm RP-1 fuel suggests the ability to detect concentrations well below 300 ppm. The range of the response is broad enough to detect low concentrations of gases (to at least 0.3%), but detection of up to 100% RP-1 fuel would require a complementary sensor as is presently done in hydrogen detection [8].

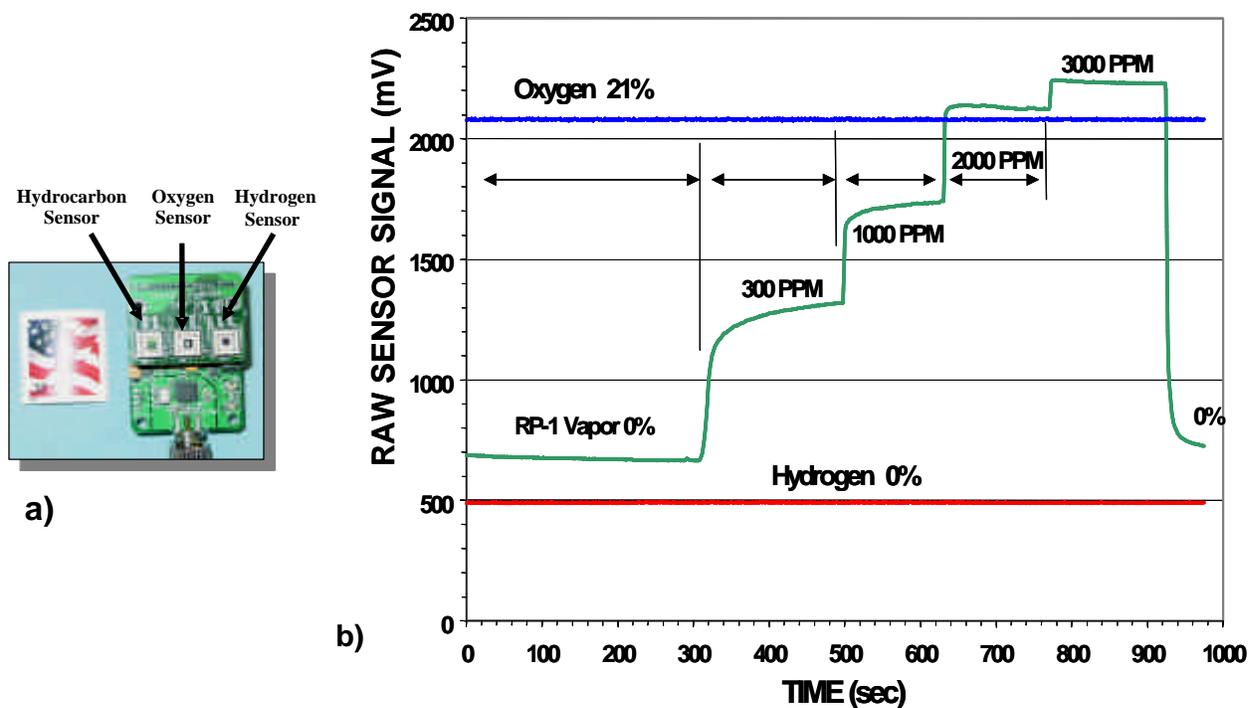


Figure 6 a) A prototype version of a “Lick and Stick” leak sensor system with hydrogen, hydrocarbon, and oxygen detection capabilities combined with supporting electronics including signal conditioning and telemetry. b) Response of the three sensors of this system to a constant oxygen environment and varying hydrocarbon (RP-1) concentrations. The sensor signal shown is the output from the signal conditioning electronics which processes the measured sensor current at a constant voltage.

This example demonstrates the use of the SiC-based Schottky diodes in applications which require high sensitivity. It also shows the ability to integrate these diodes into a complete sensor array with a full complement of supporting sensor electronics and hardware. For specific, very short term operation, like monitoring a leak for a limited time on a launch pad, this type of stability combined with high sensitivity of a properly conditioned SiC-based Schottky diode as discussed in this paper may already meet these requirements. However, frequent or long-term stable operation of the diode with high sensitivity requires further development.

SUMMARY AND CONCLUSION

SiC-based Schottky diodes have significant potential to meet the needs of a range of important aerospace applications. Reaching that full potential remains a significant technical challenge across a range of multidisciplinary technologies. A major issue is control of the surface interface between the catalytic, gas sensitive metal and the SiC semiconductor. Control of this interface will potentially enable application of the unique properties of SiC Schottky diode gas sensors. However, surface interface control of systems exposed to high temperatures and composed of a semiconductor material of maturity of SiC semiconductors is problematic.

This paper presents two examples of attempts to control the interface of a SiC-based Schottky diode gas sensor. The first method was to introduce a chrome carbide barrier layer between the catalytic metal and a defect-containing SiC semiconductor, while the second was to employ an atomically flat and abrupt SiC semiconductor surface. The chrome carbide method was demonstrated as part of a complete leak sensing system including supporting electronic hardware.

While the barrier layer approach has produced a sensitive and relatively stable sensor, the use of atomically flat SiC holds the highest potential for significantly changing the way SiC Schottky diodes are fabricated. The atomically flat approach ideally provides the same surface each time for device fabrication. This should significantly decrease issues with device reproducibility. The on-axis atomically flat surface appears to have less reactivity compared with off-axis surfaces. The initial results are quite promising but significant further testing and development still needs to be done. The development of SiC gas sensor systems is still at a relatively early level of maturity for some applications.

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